POTENTIALLY REACTIVE AGGREGATES WITH A PESSIMUM EFFECT PESSIMUM EFFECT MECHANISMS, REVIEW OF PRP QUALIFICATION TESTS AND CONDITIONS OF USE OF THESE AGGREGATES.

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Abstract

Flint and chert-rich aggregates and some other materials containing very reactive forms of silica are able to generate an AAR without giving any significant expansion and damage. These materials are called Potentially Reactive aggregate with a Pessimum effect or PRP. These aggregates give rise to gel formation but the very large number of reactive sites induces a large dispersion of this gel in the concrete. Thanks to this good dispersion, AAR gel does not lead to noticeable local stress and thus no swelling and cracking occur. These materials can be used safely in concrete structures as long as some precautions are taken. The key point is to qualify them properly before using them. The main issue with this kind of material comes from the mixture with non-reactive materials, such combinations can lead to very important swelling and concrete degradations.

Keywords: AAR, pessimum effect, gel formation, testing procedures.

1. INTRODUCTION

Most reactive silica-rich aggregates lead to concrete AAR-related degradations in humid environments when soluble alkalis are available in sufficient quantities. Paradoxically, some silica-rich aggregates containing large amounts of very reactive forms of siliceous minerals lead to very low or even no expansion at all. These aggregates, which do not generate any swelling or cracking in concrete structures in the field and no expansion in concrete prisms when tested in laboratory, can therefore, be qualified as potentially non reactive aggregates. Basically, as shown field experience, this qualification is not wrong, but because of their high proportion of reactive silica and the fact that in some circumstances these materials can nevertheless lead to significant expansions, the designation of "Potentially Reactive aggregate with a Pessimum effect" or PRP aggregates has been used to qualify them.

2. THE PESSIMUM EFFECT

With some highly and very fast reacting forms of siliceous aggregates, it has been found that the AAR-related expansion is not necessarily proportional to the content of reactive minerals in aggregates. At low proportions the expansion increases with the volume of reactive phases but beyond a certain amount the expansion decreases. With high proportions no expansion occurs at all. The proportion of reactive aggregates corresponding to the peak expansion is called the "pessimum content". With aggregates containing flint, for example, this particular situation occurs with a flint content of 20% to 30% around. When the flint proportion exceeds 60% in aggregates no expansion occurs any more. For pure opal, which is one of the most reactive forms of silica, this pessimum content is far lower, commonly between 2 and 5%.

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Figure 1 gives an example of such behaviour. In this study [1] a reactive aggregate, containing opal (around 5 wt %), was mixed in an increasing proportion with a non-reactive aggregate. From 0.5 to 2 % by mass of opal expansion gradually increased with the increase of the opal content. We find here a common situation in the AAR phenomenon. When the amount of opal phase exceeds 2.5 wt% around, the expansion begins decreasing. Beyond 4.5 wt% of opal in the aggregate, no expansion was observed any more.

This expansion curve can be divided into four main regions as a function of the opal content. In the first region (A) the reactive silica content is low and gel formation is too small to give any expansion and to induce cracking. In region (B), the amount of expansive gel is such that it can easily induce expansion and mortar or concrete cracking. In region (C), the amount of opal is higher than in the previous region but the expansion decreases with the increase of opal content. In the last region, region (D), no expansion occurs whereas the opal content is very high. In this later region, reactive sites are very numerous and well dispersed in the concrete. the gel reaction is very fast, rapidly consuming a large proportion of soluble alkalis. In this latest region, the global proportion of AAR gel is high but the proportion of gel in each reactive site is too low to give rise to expansion pressure higher than the tensile resistance of the mortar or concrete prism and thus no damage occurs. Figure 2 illustrates the comparison of expansion, surface of reactive sites, alkali content and proportion of ASR gel in each reactive site between combinations containing mixtures NR material and PR aggregates and other ones that contains mixtures of NR and PRP aggregates. Some authors [2,3] have noticed that there is a connection between the reactive SiO_2 / soluble alkali ratio and the expansion. Expansion is high as long as this ratio remains within 4 and 6 but it falls beyond 6. In the region (D) of figure 1 this ratio is likely high enough to avoid any expansion.

Aggregates containing large proportions of flint and chert show the same behaviour as mixtures with opal but with a much wider pessimum peak that makes them easier to identify. When fine and coarse aggregates used in concrete are both pure flint, there is no or very limited concrete expansion. When this kind of aggregates are mixed with non-reactive ones, leading to proportions of flint lower than 60% by a diluting effect, these combinations become potentially reactive and behave like any other common potentially reactive aggregates. By adding more non-reactive aggregates the combination can become non-reactive again if the proportion of flint drops under a level of a few percent. Thus, as long as PRP aggregates are used alone (combination of fine and coarse fractions with the same mineralogical composition) or with other potentially reactive aggregates (not necessarily with a pessimum behaviour) no expansion and damage are generally observed on mortar or concrete.

The pessimum content depends not only on the reactive silica content but also on the aggregate size (pessimum size). It has been demonstrated by some authors [4,5] that, for a given alkali content, a decrease of aggregate particle size changes the shape of the pessimum peak. This peak tends to be narrower but higher and appears for a lower reactive mineral content. This can be explained by the fact that the finer the material the larger the reactive surface area for the same percentage of reactive material. With PRP aggregates, a large fraction of initial soluble alkalis is rapidly consumed giving gel which is widely dispersed in small quantities in numerous reactive sites that does not generate expansion. The fact of decreasing the w/c ratio, so as to gain more mechanical strength for example, increases the soluble alkali content in the concrete pore solution. This alkali rise may participate in increasing gel formation. Furthermore extra alkali can be introduced from external sources, as de-icing salts, and locally raise the gel content leading to noticeable expansions. In this case, the slow diffusion of these salts into the concrete likely limits the reaction to the concrete surface.

3. FLINT AND CHERT

Chert and flint are sedimentary materials. These two materials have traditionally been confused. According to Füchtbauer and Müller [6] the term 'chert' applies to "consolidated, dense siliceous sediments with low or negligible porosity" occurring in bedded form as strata, whereas the term 'flint' is used to designate the same kind of materials but occurring in nodules or lumps. Nevertheless, after weathering, erosion, subsequent sedimentary transport and final deposition it is difficult, and often impossible, to distinguish chert from flint in sedimentary deposits. Occasionally, the white cortex, occurring along the original periphery of flint fragments may provide a clue, but in most cases it is impossible to find an unambiguous argument for one name or the other.

Chert and flint are microcrystalline rocks made of Alpha quartz, moganite (SiO_2 polymorph stable at ambient conditions) [7, 8], chalcedony and opal ($SiO_2 \bullet nH_2O$). Flint and chert are easily identified in thin section using an optical microscope equipped with rotating stage and a crossed polars illumination device. The common texture is a compact collection of micro quartz grains often mixed with small areas or beds of spherical fibrous silica (chalcedony).

4. QUALIFICATION OF PRP AGGREGATES

There are very few tests to specifically qualify potentially reactive aggregates with a pessimum effect (PRP). Most laboratory mortar bar tests or concrete prism tests, qualify aggregates as being either non-reactive (NR) or potentially reactive (PR).

4.1 Petrographic examination

Before any aggregate qualification it is strongly advised to perform a petrographic analysis. Numerous guides or standards [9 to 11] give comprehensive procedures to perform such a petrographic examination. Flint and chert are easily recognisable by visual examination. In doubt, a simple hardness test with a hammer can be done. Flint and chert are very hard rocks which give sharp lumps. In a second step, thin section can be made to identify small areas of very reactive minerals or some minerals uneasy to identify visually. Nevertheless, small proportions of opal in aggregates are often difficult to identify and localise, even on thin section. From this petrographic examination, the level of potential reactivity of aggregates can be clearly defined by a specialist. Most PRP aggregates contain very large proportions of reactive minerals as flint or chert but small amounts of very reactive minerals, as opal, can be sufficient to lead to the same behaviour..

In the Danish TI B 52 petrographic method [12], which is used only on natural sand, the material is vacuum impregnated with an epoxy resin containing a fluorescent dye. By this way porous flint grains are easily identified under a petrographic microscope with UV-light. This form of flint is known to be more reactive than dense flint.

4.2 Chemical, mortar bar and concrete prism tests

Today three main kinds of tests exist: chemical tests, mortar bar tests and concrete prism tests. As they need less heavy equipment than concrete tests mortar bar tests are widely used worldwide.

Chemical test

In France, a standardized chemical test, called the "kinetic test" (NF P18-594 and FD P18-542 [13, 14]) has been used for decades to clearly identify PRP aggregates. To perform this test, aggregates are to be crushed to get a sample containing less than 30 to 45% under 100 µm and no more than 3 to 5% sieving residue at 315 µm. This sample is then immerged for 24, 48 and 72 hours into a 1N NaOH solution at 80°C. Soluble silica and the remaining NaOH content are measured at these three times. All materials containing more than 15% carbonates must be decarbonated before testing (acid attack). The Kinetic test is not suitable for aggregates with alumina content higher than 5% (risk of silico-aluminous hydrates formation that leads to underestimate the amount of soluble silica). Thanks to the SiO2/Na2O ratio, aggregates can be qualified as non-reactive (NR), potentially reactive (PR) or potentially reactive with a pessimum effect (PRP) when the ratio is very high (Figure 3). Nevertheless, this test takes time and requires equipment as each supernatant solution must be accurately analysed, commonly by ICP.

Mortar bar tests

For years, numerous accelerated mortar bar tests have been developed all over the world, ASTM C227, C1260 and C1567 to name a few. The RILEM AAR-2 applied in the PARNER programme [15] showed that some mortar bar tests are effective at identifying pessimum behaviour, but the pessimum proportions indicated by accelerated mortar-bar tests were not necessarily the same as those exhibited in concretes using similar materials.

A few years ago, some tests were slightly modified so as to be able to better identify PRP aggregates. This identification is based on a set of mortar bars made of different aggregates/cement ratios (Table 1). In France, the Autoclave test (NF P18-594 [13]) is made on aggregate samples crushed to get a 0,160-5 mm fraction which is tested on an alkali boosted mortar (4% Na2O equivalent, 4x4x16 cm prisms) cured 5 hours in autoclave at 127°C and pressure of 0,15 MPa. By manufacturing at least 2 sets of mortar with different cement/aggregate ratios (respectively 0.5 and 1.25 and possibly 2.5) aggregates with a pessimum behaviour can be identified by comparing expansions measured with at least the first two ratios. Another mortar test, the Microbar test (NF P18-594 [13]) is common used too. In this test, aggregates are finely crushed to get a 0,160-0,630 mm fraction. This fine fraction is tested on alkali boosted mortar (1.5 % Na2O eq, 1x1x4 cm prisms). Like the previous test, several cement/aggregates ratios are used (2, 5 and possibly 10). After demoulding, mortar bars are cured 4 hours in water vapour then immerged 6 hours into a 10% KOH solution at 150°C. The expansion of the mortar bars is measured after cooling. Aggregates with a pessimum behaviour can be identified by comparing expansions measured with the first two ratios.

In these two tests, the PRP qualification is based on the fact that mortars made with the second ratio (1.25 or 5) expand less than those with the first one (0.5 or 2). With the second ratio, mortars contain 2.5 times as many aggregate as the first one but also less cement and thus less soluble alkalis. With PR aggregates, expansion tends to steadily increase from the first to the second ratio. As for PRP aggregates, the first of the two ratios commonly corresponds to the increasing slope of the pessimum peak, while the second one corresponds to the decreasing slope of this peak. Though the expansion measured with the second ratio is often smaller than the first one..

Concrete prism tests

Mortar bar tests tend to be more and more replaced by concrete prism tests either to qualify aggregates or concrete (concrete performance tests). In the aggregate qualification tests (Table 2), aggregates are tested with a fixed concrete composition. In the second case, a whole concrete composition, commonly a concrete to be used in a given concrete structure, is qualified. The first test is mainly used to qualify materials from a quarry and to provide data for a quality control follow-up while the second one is more often used to evaluate the effectiveness of preventive measures.

At the present time, the number of AAR concrete tests is limited. The most recent one, the RILEM AAR-3 test is widely based on American, Canadian, British and French tests. Basically most concrete qualification aggregates tests are based on the same kind of procedure, aggregates are not crushed and cement is alkali boosted (1 to 1,25% Na2Oeq) to shorten the testing time to around 8 to 12 months (see table 2). The main difference between these tests lies on the storage device used during the testing time. More recently, the RILEM proposed the AAR-4.1 test, which is very similar to the AAR-3.1 test but performed at 60°C instead of 38°C to shorten even more the testing period.

With these concrete tests, different kinds of combinations can be tested. Fine and coarse fractions of the same aggregates can be tested together. A fine fraction can be tested in combination with a non-reactive coarse aggregate or vice–versa. In this second case, if one of the fractions to be tested is PRP, the combination with a non reactive aggregate will be likely qualified as “PR”. This test of combinations of NR+PRP aggregates can be used to determine with several mixtures the position of the pessimum peak and the safe non expansive zone of a PRP aggregates. If two PRP fractions are tested together, the test result will be negative leading to a “NR” qualification. So as we can see, the test result can be misleading. Nevertheless the presence of large quantities of flint, chert or the presence of very reactive minerals, identified by visual examination or microscopic examination, must lead to qualify these materials as PRP if the concrete test result indicates a NR combination.

Appeared recently concrete performance tests (Table 3) qualify not a set of aggregates but the concrete itself. This new kind of test is well suited to the performance-based approach that is more and more used in different parts of the World. In France, the NF P18-454 [14] is performed on 7x7x28 cm concrete prisms stored at 60°C and 100% RH in the same container/reactor device as the NF P 18-594 (see Table 2). Here only a slight cement alkali boosting is made to take into account the Na2O eq. standard deviation of the industrial cements. The concrete composition and constituents to be tested are the same to those that will be used in the field. Performance tests are well suited to evaluate the effectiveness of preventive measures as the addition of some specific Supplementary Cementing Materials.

A new AAR-4.2 concrete performance test is still under discussion in the RILEM TC-ACS-P working group. Some points as the testing temperature, the type of container to be used are still under discussion.

4.4 General procedure to identify PRP aggregates

In France, the qualification of PRP aggregates lies on the three following steps (FD P18-542):

- Step 1: Petrography examination ➔ if flint content > 70% ➔ PRP qualification.
- Step 2: Screening tests (mortar bar tests) ➔ NR / PR or PRP qualification depending on expansion on mortar specimens manufactured with different cement / aggregate ratios.
- Step 3: Concrete test (NF P18-594) ➔ if an aggregate is qualified as NR and if its flint, jasp or radiolarit content is higher than 40 % ➔ PRP qualification.

5. FLINT AND CHERT – PRECAUSION OF USE.

As we have seen above the use of PRP aggregates is safe as long as some precautions are taken. Among other things fine or coarse PRP aggregates must not be mixed with NR aggregate otherwise this combination will be likely PR.

In the Netherlands, if the content of porous chert or flint exceeds 2 % the aggregate may be used provided that appropriate measures are taken to mitigate deleterious AAR. If the content is less than 2 %, the mortar bar test is performed to establish whether deleterious AAR can occur. In France, the LCPC guideline [16] allows the use of potentially reactive aggregates with pessimum behaviour (PRP) under two specific conditions:

- *Condition 1*: the concrete must contain fine and coarse aggregates showing both a pessimum behaviour and PRP aggregates can be mixed with PR aggregates. In the latter case, the combination must be qualified as PRP by the autoclave or the Microbar test.
- *Condition 2*: the combination of fine and coarse aggregates must contain more than 60 wt % of flint or the combination must be qualified as non reactive with the concrete test (aggregate qualification test) (threshold 0.04% at 8 months, see Table 2).

In the UK, in the 1980s, the commonly called "60% rule" was introduced in the BRE Digest 330 and Concrete Society TR30 guidance for sand plus gravel, containing more than 60% flint. This rule was in force for some years and as far as is known was effective. However, from 1990 to 1993 a comprehensive survey of sands and gravels in the UK was carried out by Rayment et al for the UK Highways Agency and that found that, in laboratory concrete prism tests, a minority of sand and gravel deposits did not follow this rule in that they showed excessive expansion despite containing more than 60% flint or chert [17]. The reason for this was that these flints or cherts were exceptionally dense and therefore presumably not sufficiently reactive. They proposed a minimum water absorption value as a way of identifying these rocks but at this stage it was decided that the application of the 60% rule was becoming too complicated and uncertain and it was decided to classify all siliceous aggregates as potentially reactive.

6. FIELD EXPERIENCES

In France, flint-rich aggregates have been widely used for ages in the Parisian Basin and in Normandy without any trouble. Flint aggregates are appreciated because of their light colour, hardness and very good resistance to freezing. As long as these flint-rich aggregates are not mixed with low or non reactive aggregates the pessimum behaviour seems to protect concrete from AAR degradation. Partly silicified limestone, chert and jasp exist in other regions, like in the Rhone valley (south east of the country), naturally mixed with non reactive materials. There, the proportion of reactive silica-rich materials does not exceed 10 to 20%, leading to major AAR-related expansion if the soluble alkali content is not limited. In the London area of the Thames valley, in the UK, there is a similar situation than in the Parisian Basin. The most commonly used aggregate combination in concrete has been a sand and gravel mixture from the Thames valley deposits used together. Both of these are flint rich. A typical Thames valley sand will contain around 25% flint, with the remainder being mainly quartz, while the gravel is typically composed of 90% flint. The resulting combination will contain 70 or 80% flint. No problems have been reported with concrete containing this combination. The greatest number of cases of AAR in the UK was in South West England in concrete in which sea dredged sand containing 60 or 70 % chert was combined with a non-siliceous coarse aggregate such as limestone or granite. The resulting combination typically contained only 15 to 30 % chert. When used in concretes with a high alkali cement (>1% Na_2O equiv.) this resulted in severe damage to many structures.

7. CONCLUSIONS

Potentially reactive aggregates with a pessimum behaviour are so intensely and quickly reactive that they are paradoxically non expansive and do not generate any trouble on concrete. This is true as long as these aggregates are used alone or mixed with PR aggregates. If they are mixed with non reactive aggregates, the combination will likely lead to expansion and damage depending on the proportion of NR aggregates. The identification of PRP aggregates lies mainly on a step by step qualification procedure. If visual or microscopical petrographic examinations reveal the presence of large quantities of flint, chert or chalcedony or more than several percents of opal and that no expansion are measured on mortar bar or concrete prisms tests made with there materials, a PRP qualification must be suspected. To support this qualification, a relevant mortar bars test, with at least two aggregates /cement ratios can be applied. Another possibility is to apply a common mortar bar or concrete prisms test and to test several mixtures containing the unknown aggregate to be qualified and different proportions of a well-known NR aggregate. In all cases this qualification process requires a good skill.

The use of PRP aggregates does not lead to any trouble as long as there are used alone or mixed with either another PRP aggregate or a PR aggregate. Pure flint has been used in some regions for decades without any trouble.

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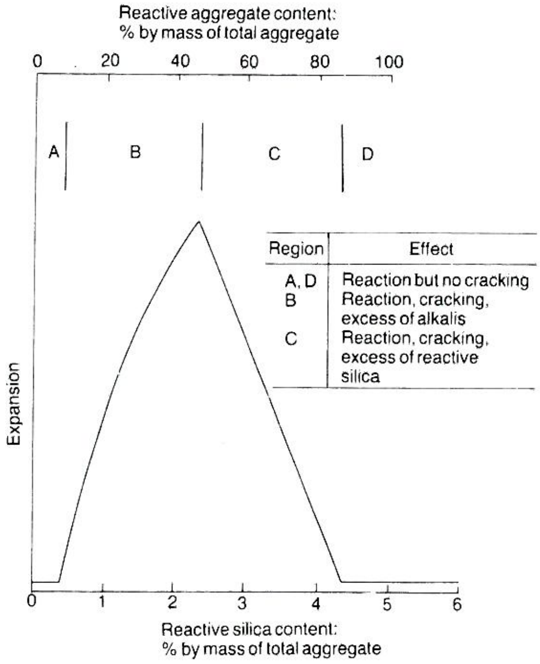

Figure 1 - The pessimum behaviour exhibited by an aggregate with opaline silica, according to [1].

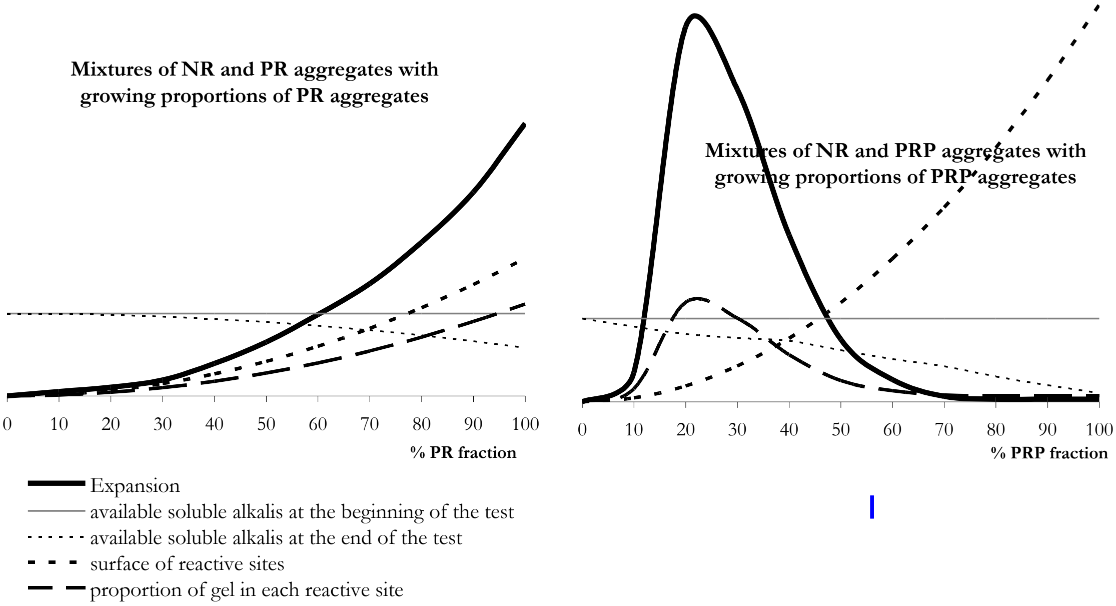

Figure 2 – Comparison of combinations of PR + NR and PRP + NR aggregates.

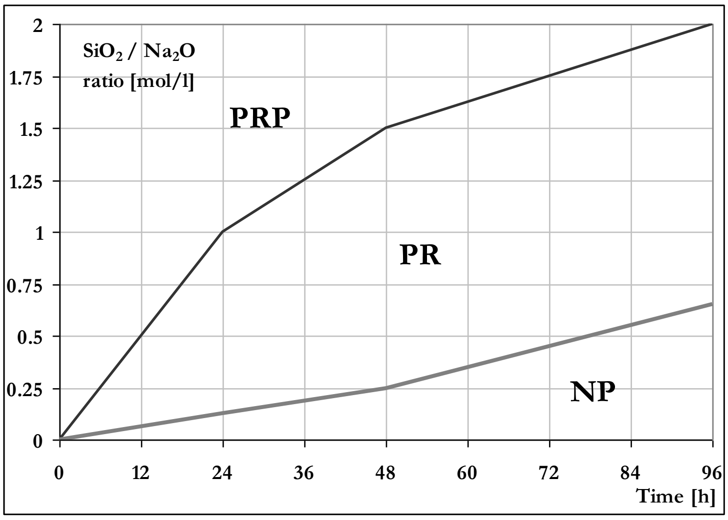

Figure 3 – Graph used for the Kinetic test to determine the level of reactivity of aggregates.

	Autoclave test (P18-494)	**Microbar test** (P18-494)
Sample range after crushing	0,063 to 5 mm	0,063 to 0,630 mm
Primes size	4x4x16 cm	1x1x4 cm
Na2O equivalent in boosted cement	**4%**	**1.50%**
Aggregates / cement ratios	0.5, 1.25, (2.5)	2, 5, (10)
Curing	5h at 127°C and 0.15 Mpa	4h in water vapor then 6h in a 10% KOH solution at 150°C
Testing time	47 hours (after demoulding)	Maximum 35 hours (after demoulding)

Qualification	Limiting values for the **Autoclave test**	
NR	Expansion lower than 0.15 %	
PRP	Expansion higher or equal to 0.15 %	Expansion on mortar with C/Agg = 1.25 is 10% higher than that with C/Agg = 0.5
PR		Not complying with NR or PRP criteria.

Qualification	Limiting values for the **Microbar test**	
NR	Expansion lower than 0.11 % for both C/Agg ratio	
PRP	Expansion higher or equal to 0.11 %	Expansion on mortar with C/Agg = 5 is 10 % higher than that with C/Agg = 2
PR		Not complying with NR or PRP criteria.

Table 1- Autoclave and Microbar tests, specificities and criteria.

	RILEM AAR-3	**BS 812-123**	**ASTM C1293**	**NF P18-594**
Sample range	no requirement	aggregates sieved into 3 fractions (0/5, 5/10, 10/20)	fraction > 19 mm crushed and incorporated into the coarse fraction	fraction > 20 mm crushed and incorporated into the coarse fraction
Cement dosage	440 kg m^3 of concrete	22% in volume	420 kg m^3 of concrete	410 kg /m^3 of concrete
Aggregates combination	40% of fine aggregates (0 to 4 mm) and 60% of coarse aggregates (4 to 22.4 mm) (standard test)	30% of 0/5, 40% of 5/10 and 30% of 10/20 fraction	coarse aggregate = 0.7 of the dry rodded bulk density of the concrete	660 Kg of fine aggregates and 1100 Kg of coarse aggregates / m3 of concrete
Free W / C ratio	0.5 in mass	1,027 in volume	0.42 to 0.45 % by mass	adjusted to get a slump of 80 +/- 20 mm
Prism dimensions	75x75x250 mm	75x75x250 mm	75x75x300 mm	70x70x280 mm
Na_2O in boosted cement	**1.25 %** (NaOH addition)	**1%** (KOH addition)	**1.25 %** (NaOH addition)	**1.25 %** (NaOH addition)
Number of prisms to be tested	3	3	3	3
Precuring period	7 days at 20°C	no	no	no
Storage device	cylindrical container with an airtight lid and a grid placed 40 mm above the bottom. The container is filled with water to a depth of 20 mm, a wick is placed around the interior wall of the container.	Concrete prisms are wrapped into a wet cotton clothe, placed into a sealed plastic bag and stored in plastic container.	Polyethylene pails with airtight lids and a performated rack at the bottom. A wick of absorbant material is placed around the inside wall from the top to the bottom. 20 mm of water above the bottom	stainless steel container with a grid placed 40 mm above the bottom. The container is filled with water to a depth of 20 mm. The container is stored in a reactor maintained at a rh of 100%
Temperature of test	**38°C**	**38°C**	**38°C**	**38°C**
Periods of measurement	2, 4, 13, 26 and 52 weeks	2, 4, 13, 26, 39 and 52 weeks	28, 56 days; 3, 6, 9 and 12 months	1, 2, 3, 4, 6 and 8 months
PR threshold	*Not fixed yet*	**0,1% at 12 months** (probably expansive)	**0.04% at 12 months**	**0.04 % at 8 months**

Table 2 – AAR concrete tests for aggregates qualification.

	NF P18-594
Sample range	Aggregates as provided by quarries (coarse aggregates ≤ 22,5 mm)
Cement dosage	the same as the field concrete to be tested
aggregates combination	the same as the field concrete to be tested
free W / C ratio	the same as the field concrete to be tested
Prism dimensions	70x70x280 mm
Na2O in boosted cement	slight cement alkali boosting to take into account the Na2O éq. standard deviation of industrial cements
number of prisms to be tested	3
precuring period	no
storage device	stainless steel container with a grid placed 40 mm above the bottom. The container is filled with water to a depth of 20 mm. The container is stored in a reactor maintained at a RH **of 100%**
Temperature of test	**60°C**
Periods of measurement	4, 8, 10, 12 weeks and more for certain types of aggregates
PR threshold	**0.02 % at 12 weeks or 5 months**

Table 3 – AAR NF P18-594 concrete performance test.